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High Quality $\text{In}_x\text{Ga}_{1-x}\text{As}$ (x : 0.08 – 0.13) Crystal Growth for Substrates of $\lambda = 1.3 \mu\text{m}$ Laser Diodes by the Travelling Liquidus-Zone Method

Kyoichi Kinoshita and Shinichi Yoda
Japan Aerospace Exploration Agency
Japan

1. Introduction

The growth of compositionally uniform alloy crystals is promising for variety of applications because lattice parameters as well as electrical and optical properties can be controlled by composition. Among them, $\text{In}_x\text{Ga}_{1-x}\text{As}$ bulk crystals are expected as substrates of laser diodes with emitting wavelength of $1.3 \mu\text{m}$. High optical gain with small temperature dependence was demonstrated for strained quantum well grown on $\text{In}_x\text{Ga}_{1-x}\text{As}$ substrates (Ishikawa, 1993; Ishikawa & Suemune, 1994). However, growth of homogeneous $\text{In}_x\text{Ga}_{1-x}\text{As}$ bulk single crystals is very difficult due to large separation of liquidus and solidus lines in the pseudobinary phase diagram (Bublik & Leikin, 1978). $\text{In}_x\text{Ga}_{1-x}\text{As}$ bulk crystals were grown by the liquid encapsulated Czochralski (LEC) method with supplying GaAs (Nakajima et al., 1991), zone levelling method (Sell, 1991) and multicomponent zone melting method (Nishijima et al. 2005). InAs mole fraction was limited to 0.2 in LEC method and zone levelling method due to temperature fluctuation in the melt and large separation of liquidus and solidus lines. Multicomponent zone growth (MCZG) using a seed with graded InAs concentration produced a single crystal with InAs mole fraction of 0.3 and length of about 17 mm (Nishijima et al. 2005). However, MCZG requires complicated growth technique and no good reproducible results were obtained. In all of the methods, the most difficult point is to keep the freezing interface temperature constant for growing compositionally uniform crystals since interface travelling rate depends on temperature gradient, mass transport in a melt and solute concentration gradient ahead the interface. As a result, no device quality $\text{In}_x\text{Ga}_{1-x}\text{As}$ crystals with uniform composition have been obtained so far.

The travelling liquidus-zone (TLZ) method was invented for keeping the interface temperature constant and growing compositionally uniform alloy crystals (Kinoshita et al., 2001, Kinoshita et al., 2004). In the TLZ method, the interface travelling rate can be determined exactly if temperature gradient in the zone is known and then the interface position can be fixed in relation to the heater position by translating a sample device in accordance with the interface travelling rate (Kinoshita et al. 2002). Principle of the TLZ method is proven by the precise measurement of temperature gradient in the melt zone,

growth rate and the composition of grown crystals (Nakamura et al. 2003). Since the TLZ method requires diffusion limited mass transport, diameter or thickness of the melt is limited to small values on the ground for suppressing convection in a melt. 2 mm thick platy $\text{In}_x\text{Ga}_{1-x}\text{As}$ (x : 0.1 – 0.13) crystals were grown by the TLZ method as substrates of laser diodes with $\lambda = 1.3 \mu\text{m}$ (Kinoshita et al. 2008). Quality of grown crystals was characterized by electron probe micro analyzer (EPMA), electron back scattering pattern (EBSP), X-ray diffraction (XRD) and by fabricating laser diodes. High temperature stability of output power of laser diodes was proven (Arai et al. 2009).

In this chapter, principle of the TLZ method, growth of high quality $\text{In}_x\text{Ga}_{1-x}\text{As}$ platy crystals, fabrication of laser diodes with $\lambda = 1.3 \mu\text{m}$ and characterization of laser diodes are reviewed.

2. TLZ method

The TLZ method was originally invented for compositionally uniform alloy crystal growth in microgravity. Principle of the TLZ method is studied by growing small diameter $\text{In}_x\text{Ga}_{1-x}\text{As}$ crystals on the ground (Kinoshita et al., 2002, 2003, Nakamura et al., 2003, Kinoshita and Yoda, 2010) because convection in a melt can be suppressed in capillary tubes even on the ground. In this section, principle of the TLZ method, one dimensional model and evaluation of a model are reported.

2.1 Principle of a TLZ method

It is well known that compositionally uniform alloy crystals can be grown if diffusion controlled steady-state growth is achieved (Tiller et al., 1953). However, even if such growth is realized during directional solidification, initial transient region is inevitable and long crystal length is required to reach the steady-state. Moreover, growth rate should be sufficiently high because solute in the boundary layer diffuses out and solute should be supplied to keep constant solute concentration at the interface. Segregation at solidification supplies solute and amount of supplied solute is determined by growth rate. This is the reason why sufficiently high growth rate is required for the steady-state growth. High growth rate, however, tends to cause the constitutional supercooling (Tiller et al., 1953). Many investigators tried to grow compositionally uniform alloy crystals by diffusion controlled steady-state growth but none of them obtained satisfactory results. Microgravity experiments are also expected to realize diffusion controlled steady-state growth by suppressing convection in a melt but no one succeeded in growing homogeneous alloy crystals having expected composition. Residual gravity in space crafts on the order of $10^{-4} g$ hinders diffusion controlled growth. Therefore, a new growth method for obtaining compositionally uniform alloy crystals is anticipated.

When we consider diffusion boundary layer in the steady-state growth, we noticed that zone melting method under temperature gradient is easier to maintain constant solute concentration at the interface because solute is saturated at freezing and dissolving interfaces. Moreover, solute is almost saturated throughout the zone if zone thickness is thin enough. As described later in more detail, in such situation solute concentration gradient in the zone is controlled by temperature gradient and compositionally uniform alloy crystal growth becomes much easier than other growth method. We named this zone melting method under temperature gradient a travelling liquidus-zone (TLZ) method since crystal

growth is carried out by travelling almost saturated liquid-zone. Sample configuration, temperature profile, solute profile in a sample and its relation to a phase diagram in the TLZ method is depicted in Fig. 1 as referring to the growth of $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$. It is needless to say that convection in a thin melt zone is suppressed more effectively than in a long melt even in microgravity.

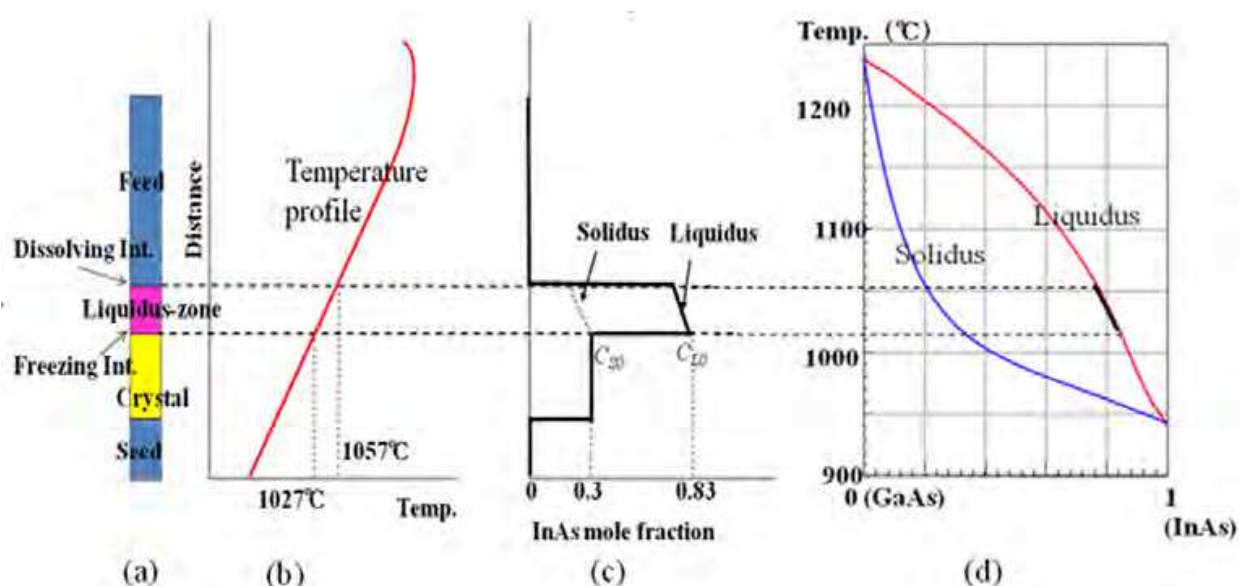


Fig. 1. Schematic drawing of a TLZ method as referring to the growth of $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$. (a) sample configuration, (b) temperature profile, (c) InAs concentration profile, (d) GaAs-InAs pseudobinary phase diagram.

In Fig. 2, solute concentration profile in the diffusion boundary layer in the steady-state growth is compared between directional solidification method and TLZ method (Kinoshita and Yoda, 2011). When temperature gradient is low and zone thickness is thin, solute concentration profile is linearly approximated and it can be related to the liquidus line in the pseudo binary phase diagram as shown in Fig. 1. Liquidus line is also linearly approximated in a narrow temperature range. Then, it should be noticed that solute concentration gradient in a zone can be controlled by temperature gradient in such case as is given by eq. (1), where C_L is solute concentration in the zone, T is temperature, Z is a distance measured from the freezing interface and $\partial C_L / \partial T$ is reciprocal of the slope of the liquidus line in the phase diagram. Then, freezing rate V is given by eq. (3) if we suppose the diffusion controlled steady-state growth is realized as given by eq. (2) and V is determined by temperature gradient in the zone, where D is interdiffusion coefficient between solute and solvent, C_{L0} and C_{S0} are liquidus and solidus concentrations at the freezing interface.

$$\left(\frac{\partial C_L}{\partial Z} \right)_{Z=0} = \left(\frac{\partial C_L}{\partial T} \right) \left(\frac{\partial T}{\partial Z} \right)_{Z=0} \quad (1)$$

$$V(C_{L0} - C_{S0}) = -D \left(\frac{\partial C_L}{\partial Z} \right)_{Z=0} \quad (2)$$

$$V = -\frac{D}{(C_{L0} - C_{S0})} \left(\frac{\partial C_L}{\partial Z} \right)_{Z=0} = -\frac{D}{(C_{L0} - C_{S0})} \left(\frac{\partial C_L}{\partial T} \right) \left(\frac{\partial T}{\partial Z} \right)_{Z=0}$$

(3)

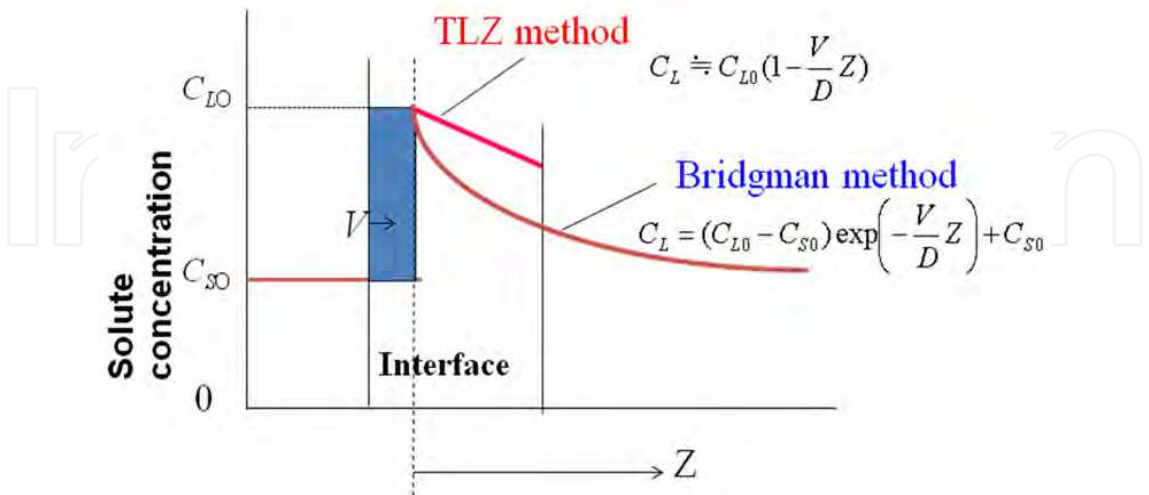


Fig. 2. Comparison of solute concentration profile between directional solidification (Bridgman method) and zone melting method under temperature gradient (TLZ method).

If we know interdiffusion coefficient D and phase diagram data, we can calculate the freezing rate V using eq. (3). Then, it is very easy to fix the interface position as shown in Fig. 3. If a sample device is translated toward opposite direction at the same rate of interface travel rate as calculated by eq. (3), the interface position is fixed and the interface temperature is kept constant, resulting in a compositionally uniform alloy crystals. Thus, compositionally uniform alloy crystals are grown by simply translating a sample device at constant rate in the TLZ method. According to our numerical analysis, 1 to 2 hours are required for establishing an almost saturated solute concentration profile in a zone and spontaneous crystal growth starts.

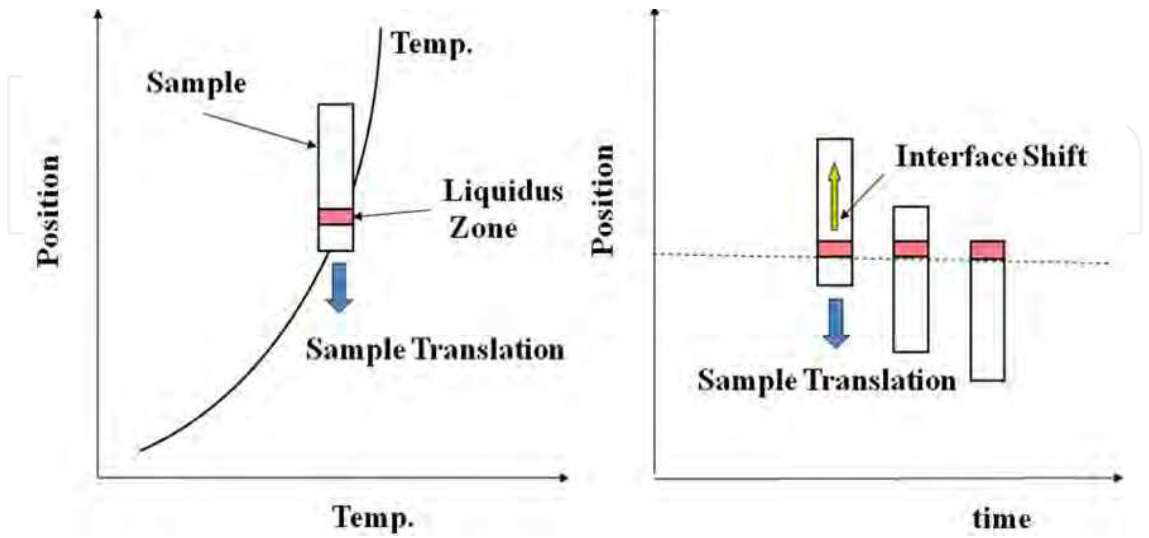


Fig. 3. Schematic view of a liquidus-zone shift in the TLZ method and a way to keep the freezing interface at a fixed position.

2.2 Evaluation of a TLZ growth model

One dimensional TLZ model equation (3) is evaluated by growth experiments. A seed, a zone forming material and a feed were inserted into a boron nitride crucible and the crucible was vacuum sealed in a quartz ampoule at about 1×10^{-4} Pa. In the growth of $\text{In}_x\text{Ga}_{1-x}\text{As}$, the zone forming material is InAs or InAs rich solid solution with GaAs. The ampoule was inserted into a temperature gradient furnace and was heated to about 1100°C at temperature gradients of $10 - 20^\circ\text{C}/\text{cm}$. InAs-GaAs interdiffusion coefficient was measured in microgravity using a sounding rocket (Kinoshita et al. 2000). It was measured to be $1.5 \pm 0.2 \times 10^{-8} \text{ m}^2/\text{s}$ at 1070°C . For $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$ growth, C_{L0} is given by 0.83 from phase diagram. $\partial C_L / \partial T$ is also given by phase diagram as -465 K/mol . Then, V is calculated to be 0.22 mm/h for the temperature gradient of $10^\circ\text{C}/\text{cm}$ (Nakamura et al., 2003). Crystal growth experiments were performed for crystals with 2 mm diameter in order to suppress convection in a melt on the ground. Concentration profiles were measured by EPMA. Results are shown in Fig. 4. In the figure, crystal growth started at the length of about 8 mm and the sample was quenched at the length of about 32 mm. Therefore, about 24 mm long crystal was grown. It should be noted that expected InAs concentration (15 at%) is realized without initial transient region. This is one unique point typical to the TLZ method when compared with diffusion controlled steady-state growth in the directional solidification. This is because steady-state solute concentration profile can be established without transient region in the case of zone melting method under temperature gradient. Compositional homogeneity is excellent; In concentration $15 \pm 0.5 \text{ at\%}$ is achieved for the length of more than 20 mm. Scattered concentration region was liquidus-zone before quenching. Dendrite crystals were grown during quenching and dendrite growth resulted in concentration scattering.

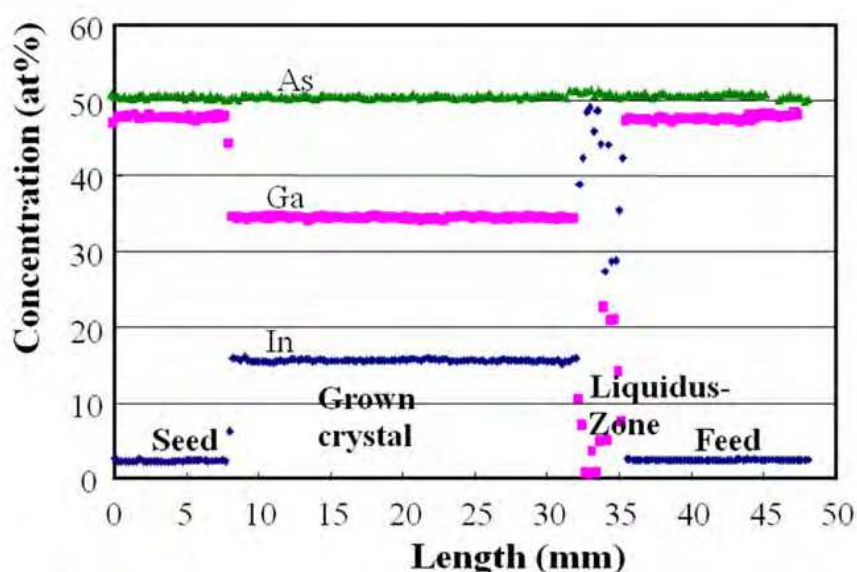


Fig. 4. Axial In, Ga and As concentration profiles for a TLZ-grown 2 mm diameter crystal at a sample translation rate $R = 0.22 \text{ mm/h}$.

It is astonishing that excellent compositional homogeneity was achieved at just calculated sample translation rate using eq. (3). Since temperature gradient in the zone was measured precisely by sophisticated method namely by measuring solidus compositions at two

interfaces (freezing and dissolving interfaces), model evaluation was possible. Solidus compositions measured at two interfaces are shown in Fig. 5 together with a determined temperature gradient in the zone (Nakamura et al., 2003, Kinoshita et al., 2005). At the freezing interface, In concentration was 15.1 at% and this concentration gives solidus temperature of 1083.1°C from the phase diagram, while In concentration at dissolving interface 12.3 at% gives solidus temperature of 1099.8°C. Distance between two interfaces is 16.8 mm and temperature gradient 10°C/cm was determined. In the temperature gradient measurement, a special sample whose In concentration is gradually decreasing towards a feed and solid-liquid coexistence at the dissolving interface was used. This enabled us to measure solidus composition at the dissolving interface.

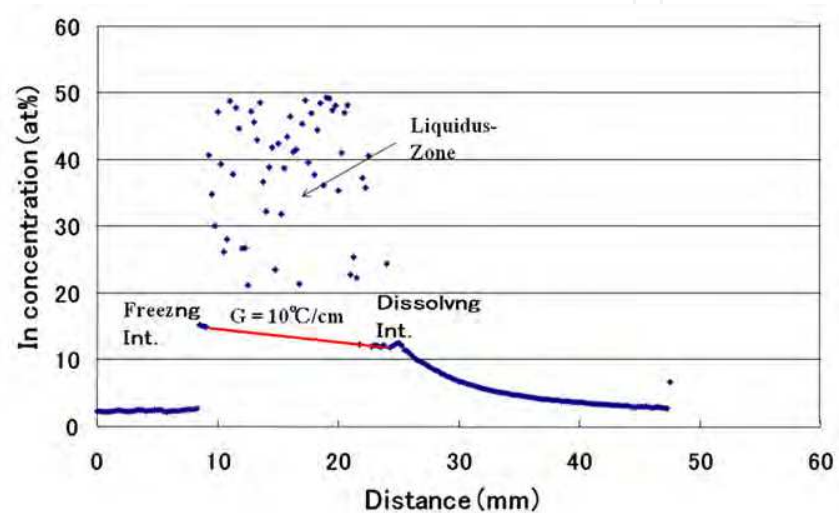


Fig. 5. Measured solidus In concentrations at freezing and dissolving interfaces and determination of temperature gradient (*G*) in the melt zone.

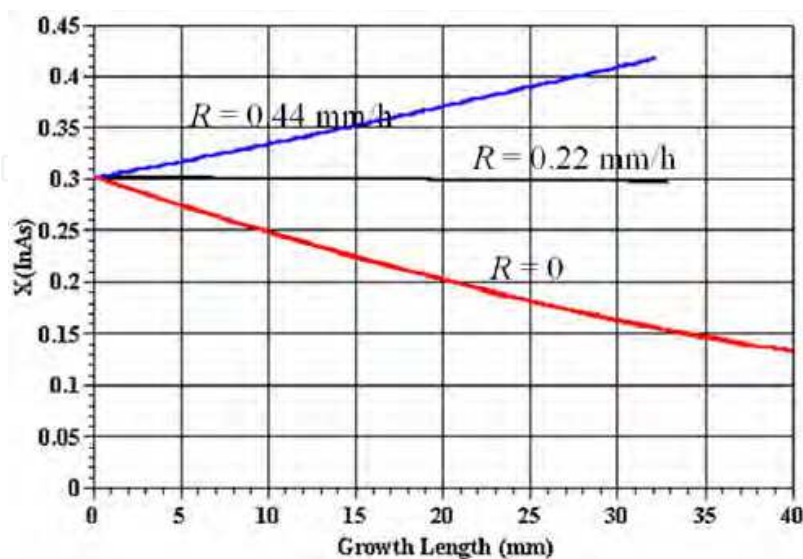


Fig. 6. Results of numerical analysis of axial InAs concentration in mole concentration as a parameter of sample translation rate *R*.

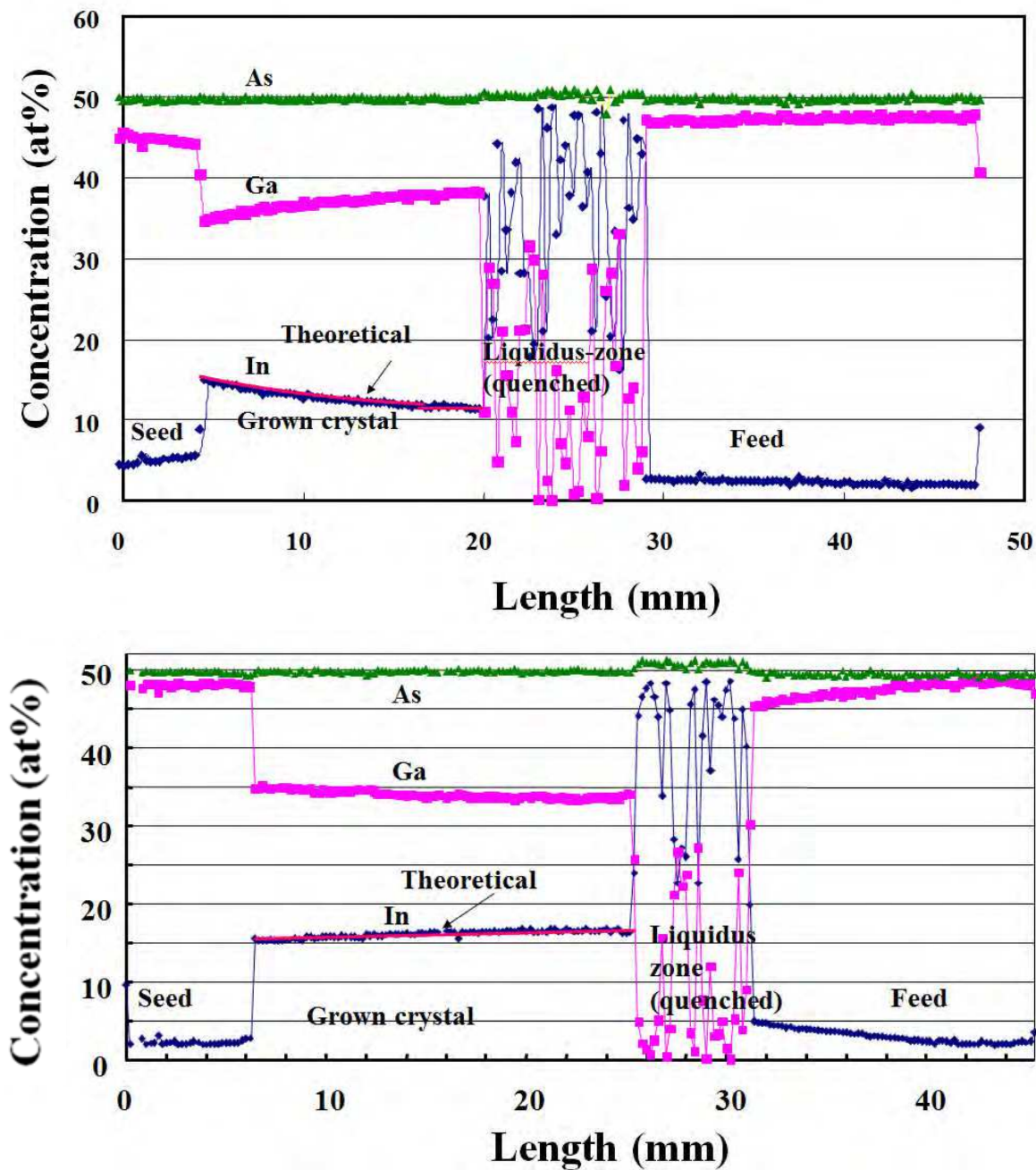


Fig. 7. Axial compositional profiles of TLZ-grown $\text{In}_x\text{Ga}_{1-x}\text{As}$ crystals at a sample translation rate (a) $R = 0$ and (b) $R = 0.27 \text{ mm/h}$.

Numerical analyses show that InAs concentration increases when sample translation rate is faster than the predicted one from eq. (3), while it decreases when sample translation rate is slower than the predicted one (Fig. 6). It should be noted that spontaneous growth occurs even if sample is stationary. This is because solute concentration difference is established between the two interfaces and interdiffusion occurs in a melt zone. At the freezing interface, In concentration is higher and In is transported towards the dissolving interface by diffusion. Indium concentration decrease causes solidification at the freezing interface because equilibrium temperature gets higher. However solidification segregates In at the

interface. The segregated In is diffused away towards the dissolving interface. At the dissolving interface, a feed is dissolved by transported In because In concentration gets higher than the equilibrium one. Thus, crystal grows at the freezing interface and a feed is dissolved at the dissolving interface; crystal growth proceeds spontaneously towards lower In concentration side (dissolving interface side, which is a higher temperature side). Sample translation towards a lower temperature side plays a role of keeping the interface temperature constant and it is not a driving force for crystal growth. The driving force in the TLZ growth is interdiffusion between solute and solvent in a zone and segregation on solidification. This should be noted clearly as one of features of the TLZ growth. Figures 7 (a) and 7 (b) show experimental results when sample translation rates do not match the freezing rate of 0.22 mm/h at a temperature gradient of 10°C/cm (Kinoshita et al., 2005). Fig. 7 (a) is a case of $R = 0$ (stationary) and Fig. 7 (b) is a case where R is faster than 0.22 mm/h. Both results agree well with the numerical analyses. As described later, higher sample translation rate than 0.3 mm/h caused constitutional supercooling and resulted in fluctuation of composition. Therefore, in Fig. 7 (b) an experimental result at $R = 0.27$ mm/h is compared with a numerical analysis. Thus, TLZ growth model was confirmed by growth experiments at various sample translation rates and comparison of compositional profiles with those obtained by numerical analyses. Latent heat effect on growth rate was also investigated by numerical analysis and the effect is shown to be negligibly small except for end member region (Adachi et al., 2004b).

2.3 Limitation in the TLZ method

TLZ growth model is established when solute concentration gradient in the melt zone is linearly approximated as shown in Fig. 2. When temperature gradient is low and zone thickness is thin, such approximation is valid. However, freezing rates deviate from those predicted by eq. (3) at high temperature gradient and for long zone length. Amount of solute in a zone also limits the length of a grown crystal. Moreover, TLZ growth model is based on the diffusion controlled steady-state growth and compositional uniformity is affected by convection in a melt. The TLZ method also lies side-by-side with constitutional supercooling since almost saturated melt is utilized. In this section, such limitations will be described in more detail.

2.3.1 Limitation by temperature gradient

TLZ method is classified as diffusion controlled steady-state growth method as well as a zone method. Solute concentration profile in the diffusion boundary layer is expressed as eq. (4) (Tiller et al., 1953). When VZ/D is small, concentration gradient is approximated by linear relation as eq. (5). Such situation has already been described in Fig. 2. Low temperature gradient gives small value of V and gives a small error in growth rate calculation using eq. (3). However, when temperature gradient is high, V gets large and an error in growth rate calculation becomes large (Adachi et al. 2004a). Figure 8 shows deviation of growth rate from linear relation to temperature gradient. From Fig. 8, it can be said that TLZ growth rate defined by eq. (3) is valid within 10% error when temperature gradient is smaller than 40°C/cm for the zone thickness of 20 mm. Therefore, TLZ growth should be carried out at low temperature gradient like 10 - 20°C/cm at zone thickness of 20 mm.

$$C_L = (C_{L0} - C_{S0}) \exp\left(-\frac{V}{D}Z\right) + C_{S0}$$

(4)

$$C_L = C_{L0} \left(1 - \frac{V}{D}Z\right)$$

(5)

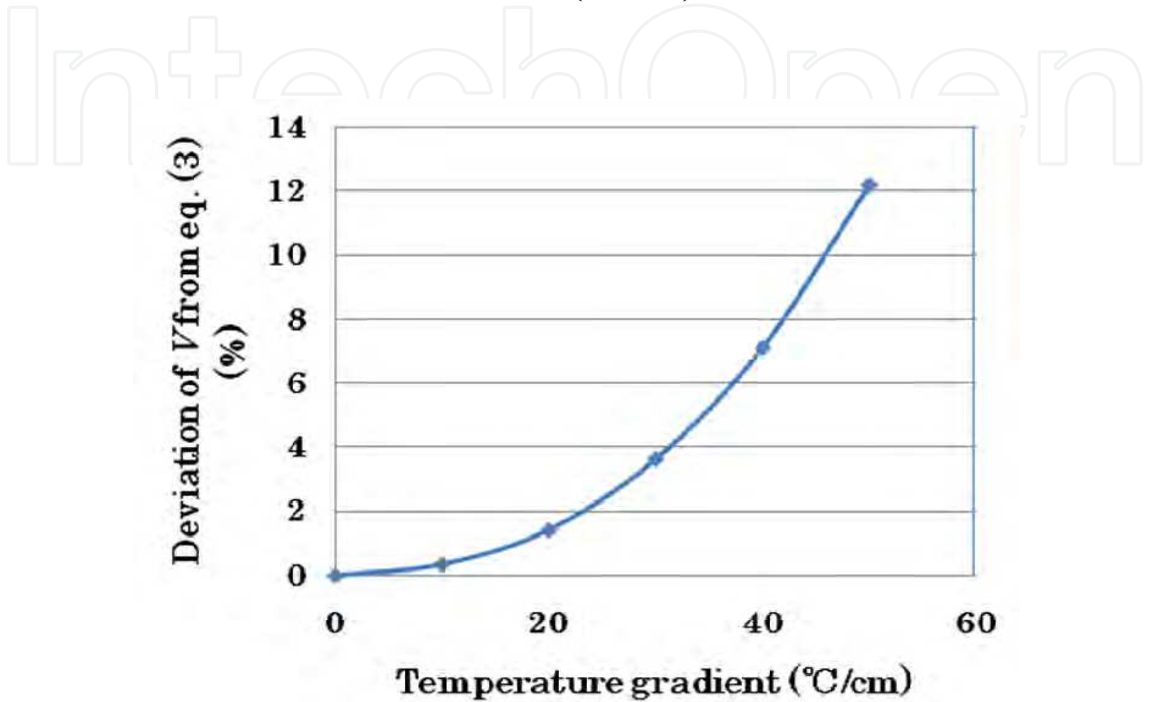


Fig. 8. Deviation of V from eq. (3) as a function of temperature gradient.

2.3.2 Limitation by zone length

Distance Z is also a variable in eq. (4). Therefore, zone length gives similar effect in growth rate calculation. When the temperature gradient is 20°C/cm, an error in the growth rate calculation using eq. (3) is about 7 % for the zone length of 40 mm but it beyonds 12 % when the zone length is 50 mm. If growth rate deviation from linearity is desired less than 10%, zone length should be less than 45 mm. Limitation of zone length is natural since long zone length cannot be distinguished from directional solidification.

2.3.3 Limitation of growth length

In the In_xGa_{1-x}As crystal growth, we usually start from a combination of a GaAs seed, an InAs zone forming material and a GaAs feed. GaAs saturated InAs rich zone is formed and this zone travel towards a GaAs feed. Therefore, InAs is consumed according as crystal growth proceeds. If an InAs length is 10 mm, length of a grown crystal is limited to 10/3 mm for an In_{0.3}Ga_{0.7}As crystal. In order to increase the length of grown crystal longer zone or InAs contained feed should be used. However, zone length is limited from the point of zone growth as described above and in an InAs contained feed InAs content is restricted to low for preventing melting of a feed. In this meaning, the length of a TLZ-grown crystal is limited.

2.3.4 Limitation by convection in a melt

The TLZ method is possible in the diffusion limited regime. If convection occurs in a melt, solute concentration profile ahead the interface is disturbed and the growth rate does not obey eq. (3). We grew 2 mm diameter $\text{In}_x\text{Ga}_{1-x}\text{As}$ crystals by suppressing convection in a melt and confirmed the TLZ growth model. However, 2 mm diameter crystals cannot be used for opt-electronic devices and we tried increase of crystal diameter. Result of a growth experiment for 10 mm diameter crystal is shown in Fig. 9. Axial compositional homogeneity is achieved for the length of more than 30 mm but radial concentration inhomogeneity exists in a 10 mm diameter crystal. According to numerical analysis, convective vortices occur at both freezing and dissolving interfaces as shown in Fig. 10. When such convective vortices are taken into consideration, radial concentration difference is understood; In-rich melt is transported towards the centre by convection and InAs concentration increases in the centre of a grown crystal. As for the strength of convective driving force, Grashof number G defined by eq. (6) gives order of magnitude where g is gravity acceleration, β is volume expansion coefficient of a melt, ΔT is a temperature difference between the two ends of a melt, L is characteristic length of a melt and ν is kinematic viscosity of a melt. Comparison of convective driving force between 2 and 10 mm diameter melts, convection in 10 mm diameter melt gives 2 orders of magnitude higher than that in 2 mm diameter melt. Result in Fig. 9 shows importance of suppressing convection in a melt for compositionally homogeneous alloy crystals in radial direction as well as in axial direction (Kinoshita et al., 2006).

$$G_r = \frac{g\beta\Delta TL^3}{\nu^2} \quad (6)$$

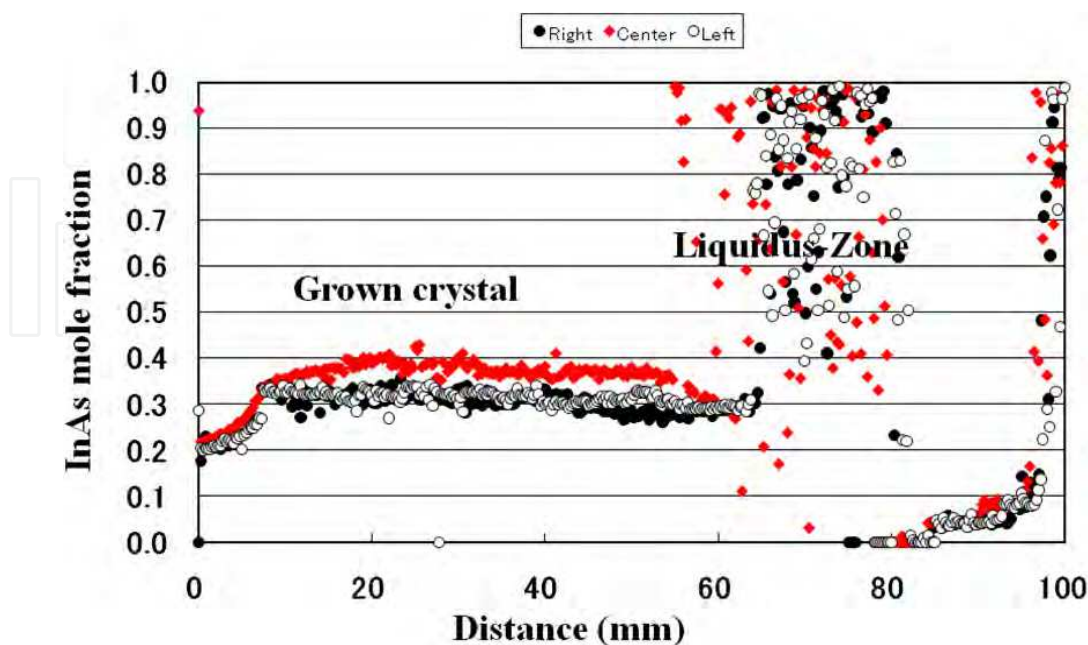


Fig. 9. Axial InAs concentration profiles compared among centre line, right and left peripheral regions.

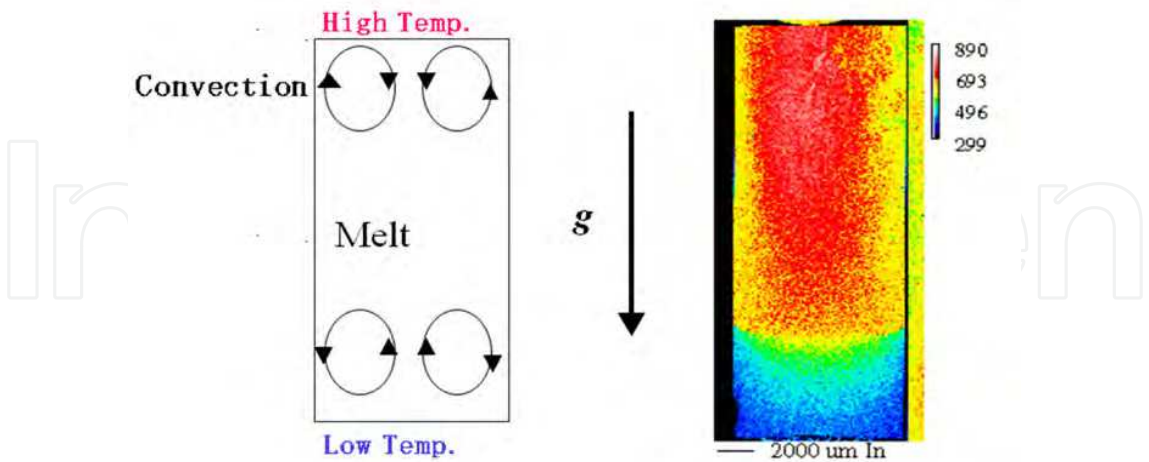


Fig. 10. Convective vortices in a melt and two dimensional In concentration map for a 10 mm diameter crystal.

2.3.5 Limitation by constitutional supercooling

The TLZ growth utilizes almost saturated melt. In this point of view, constitutional supercooling tends to occur. Constitutional supercooling in the directional solidification of alloy crystals in the diffusion limited growth was studied extensively by Tiller et al. (1953). According to them, the growth rate should be high and should fulfil conditions defined by eq. (7) where G is temperature gradient, R is growth rate, m is the slope of the liquidus line. The TLZ method utilizes solute concentration gradient in diffusion boundary layer ahead the freezing interface. Therefore, growth rate should fulfil the conditions proposed by Tiller et al. When we change notation of eq. (3) to similar ones given by Tiller et al. for comparison, equation (8) is given. Equation (8) is further modified into eq. (9). When we compare eq. (7) with eq. (9), we notice that the growth rate for realizing compositional uniformity in the TLZ method is the critical growth rate for avoiding constitutional supercooling. If growth rate is higher than this, constitutional supercooling will occur. From this discussion, it is clearly shown that homogeneous crystal growth in the TLZ method is side-by-side the constitutional supercooling.

$$\frac{G}{R} \geq \frac{m(C_{L0} - C_{S0})}{D} \tag{7}$$

$$R = \frac{D}{(C_{L0} - C_{S0})} mG \tag{8}$$

$$\frac{G}{R} = \frac{m(C_{L0} - C_{S0})}{D} \tag{9}$$

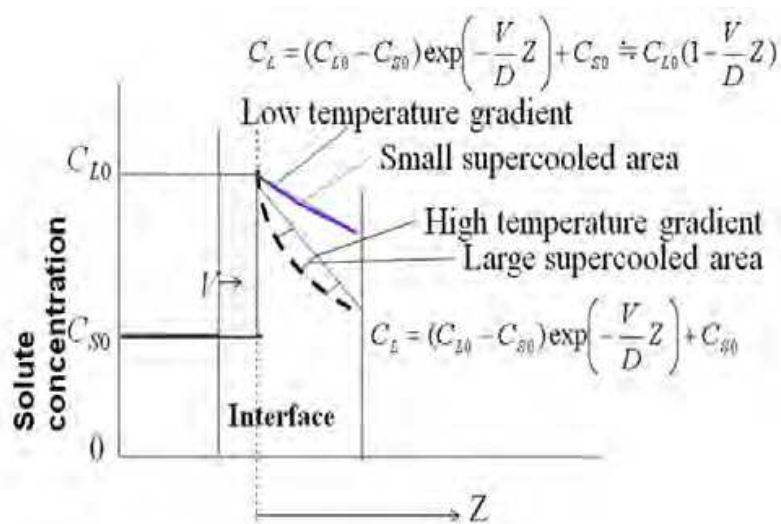


Fig. 11a. Comparison of constitutionally supercooled area during TLZ growth at low and high temperature gradients. At high temperature gradient, non-linearity of solute concentration profile ahead the freezing interface is large and gives larger supercooled area

Constitutional supercooling is inevitable at high temperature gradient (Kinoshita and Yoda, 2011). This may be peculiar from the requirement of high G/R ratio shown by eq. (7) but this requirement comes from constant G/R for compositional uniformity and solute saturation at two interfaces (freezing and dissolving interfaces) in the TLZ method. At high temperature gradient, non linearity of solute concentration profile increases as shown in Fig. 11(a). In the figure, the straight line shows equilibrium concentration at the given temperature gradient and dashed region is constitutionally supercooled region. It should be noted that constitutionally supercooled area is larger at higher temperature gradient. This is unique point in the TLZ method. It is obvious that degree of constitutional supercooling is greater at higher temperature gradient because deviation from linearity in solute concentration profile is greater at higher temperature gradient (Kinoshita et al., 2011). From this point of view, temperature gradient is limited and growth rate in turn is also limited. In the $\text{In}_x\text{Ga}_{1-x}\text{As}$ crystal growth experiments, it is shown that temperature gradient that exceeds $30^\circ\text{C}/\text{cm}$ resulted in constitutional supercooling. This means that growth rate is limited to 0.66 mm/h . In general, compositional fluctuation increases in constitutionally supercooled region. An example of compositional profile of constitutionally supercooled sample is shown in platy crystal growth in later section (an $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ platy crystal grown at a temperature gradient of $37^\circ\text{C}/\text{cm}$ showed large compositional fluctuation as shown in 3.2.3).

3. Platy crystal growth

For device application, large surface area is required. However, increase in crystal size increases convection in a melt and compositional uniformity of TLZ-grown crystals is deteriorated. Suppressing convection in a melt and large surface area is fulfilled by the growth of platy crystals if thickness of the platy crystal is sufficiently thin (Kinoshita et al., 2008, 2010). Such situation is schematically depicted in Fig. 11(b). We have experimentally confirmed that convection in a 2 mm diameter melt is suppressed and TLZ mode crystal growth is possible. Then, we determined thickness of platy crystals as 2 mm. In this section, growth and characterization of platy crystals for substrates of laser diodes with emitting wavelength $\lambda = 1.3 \mu\text{m}$ are described.

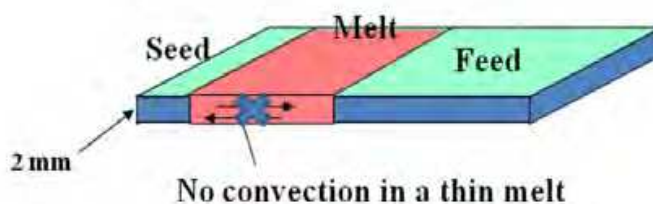


Fig. 11b. Schematic view of a platy crystal growth.

3.1 Growth procedures and characterization methods

Growth method for platy crystals is similar to that of the cylindrical crystal. A GaAs seed, an InAs zone forming material, and a GaAs feed were cut into plates with 2 mm thickness and were inserted into a boron nitride crucible with a rectangular bore and were sealed in vacuum in a quartz ampoule and then heated in a gradient heating furnace. Growth interface temperature was adjusted around 1100°C so that $\text{In}_x\text{Ga}_{1-x}\text{As}$ (x : 0.1 – 0.15) crystals were grown. Temperature gradient was set around 20°C so that constitutional supercooling at high temperature gradient was avoided. Samples were translated at the rate calculated using eq. (3). Seed orientation was $\langle 100 \rangle$ or $\langle 110 \rangle$. Sn was doped as n-type dopant. At the beginning of crystal growth, we thought that $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$ composition was required as substrates for $\lambda = 1.3 \mu\text{m}$ laser diodes. However, it turned out that composition around $\text{In}_{0.1}\text{Ga}_{0.9}\text{As}$ can be used as substrates since thin film growth technology has been developed and large lattice mismatch between the substrate is conquered. As for crystal size, we started from the growth of 10 mm width and increased crystal width. At the present, we are successful in growing 50 mm wide platy crystals. Grown crystals were polished and crystalline nature was investigated. Compositional profiles of grown crystals were measured by EPMA on polished surfaces. Two-dimensional semi-quantitative mapping analysis was performed for measuring overall compositional distribution. Quantitative analysis was performed along growth axis with precision of 0.1 at% for each constituent element. Crystal quality was evaluated by measuring X-ray rocking curves. Electrical properties of crystals were measured by Hall measurements. Etch pit density was measured after KOH etching.

3.2 Results and discussion

Experimental results for platy crystal growth are summarized and factors that effect on crystal quality are discussed in this section. Factors for affecting crystal quality are common for the TLZ method except for seeding in rectangular crucible.

3.2.1 Single crystalline nature

Typical two examples of roughly polished surface of two platy crystals are shown in Fig. 12. Composition of Fig. 12 (a) is $\text{In}_{0.10}\text{Ga}_{0.90}\text{As}$ and that of Fig. 12 (b) is $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$. When InAs mole fraction in a grown crystal is less than 0.1, a single crystal that takes over the seed orientation was grown. However, when InAs mole fraction is more than 0.13, single crystal growth that takes over the seed orientation became very difficult. This may be due to increase in lattice mismatch between a GaAs seed and a grown crystal. Difficulty in single crystallization was settled by using a feed having the same orientation of a seed. In Fig. 12 (b), polycrystallization is observed at the seed and grown crystal interface. However, a single crystal was grown again as crystal growth proceeded.

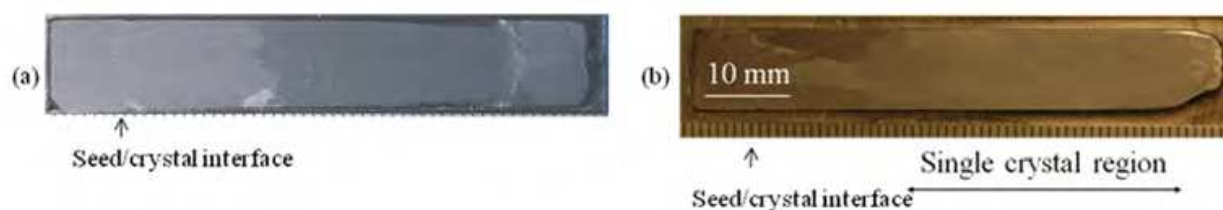


Fig. 12. Roughly polished surface of two platy crystals, (a) $\text{In}_{0.10}\text{Ga}_{0.90}\text{As}$ and (b) $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$.

The mechanism of single crystal growth is not clear but it may be related to a crucible shape having rectangular bore and nucleation of the same orientation as a feed might occur in a narrow gap between the crucible wall and feed surface. In wider crystal growth too, single crystals were grown by the same mechanism. Reproducibility of single crystal growth was very good and single crystal length more than 50 mm was obtained.

3.2.2 Growth temperature

Freezing interface temperature determines composition of grown crystals. In this point of view, growth temperature is very important. In the TLZ method, solute in a melt is almost saturated and it is very easy to control interface temperature. We set 8 thermocouples around a growth ampoule and one of them is set at seed/zone interface. Only procedure is to monitor temperatures measured by these thermocouples and to change the interface temperature. It is usual that surface temperatures of a quartz ampoule are not equal to inner temperatures of a crucible but difference between the two was less than 5°C and minor adjustment gave target compositions. Figure 13(a) is an example of $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ crystal and Fig. 13(b) is an example of $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ crystal. In $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$, interface temperature was set at 1057°C and in $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ it was set at 1095°C . In both cases, target compositions were obtained at the seed/crystal interface and no initial transient regions were found as is the case of $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$ (Fig. 4). Compositional uniformity was excellent for $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$; InAs mole fraction was 0.13 ± 0.005 , but uniformity was degraded for $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$. Single crystallinity was also better for lower In concentration crystals. Since we succeeded in laser operation at the wavelength of $1.3 \mu\text{m}$ for laser diodes on $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ substrates (Arai et al, 2007), we improved crystal quality for the composition of around $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$.

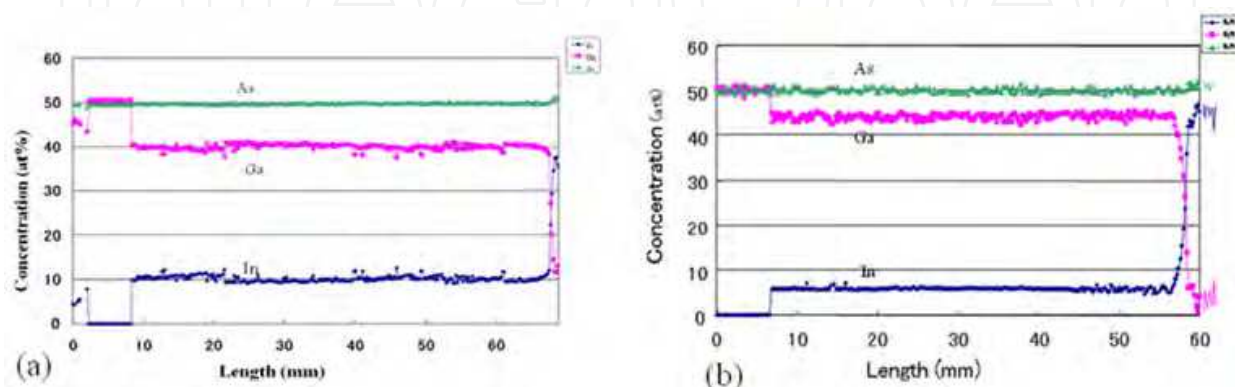


Fig. 13. Axial concentration profiles of platy crystals; (a) for $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ and (b) for $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$.

3.2.3 Temperature gradient

Temperature gradient determines spontaneous growth rate in the TLZ method as given by eq. (3). So long as the sample translation rate matches this spontaneous growth rate, compositionally uniform alloy crystals can be grown. However, high temperature gradient causes constitutional supercooling and results in compositional fluctuation. Figure 14 compares compositional profiles of crystals grown at different temperature gradient (a) $25^\circ\text{C}/\text{m}$ and (b) $37^\circ\text{C}/\text{cm}$. Note that the crystal grown at lower temperature gradient shows lower compositional fluctuation. As described in the section 2.3.5, maximum temperature gradient for suppressing constitutional supercooling may be $30^\circ\text{C}/\text{cm}$. It is concluded that temperature gradient should be as low as possible for suppressing compositional fluctuation but the lowest limit of temperature gradient should be determined from the point of mass productivity of crystals.

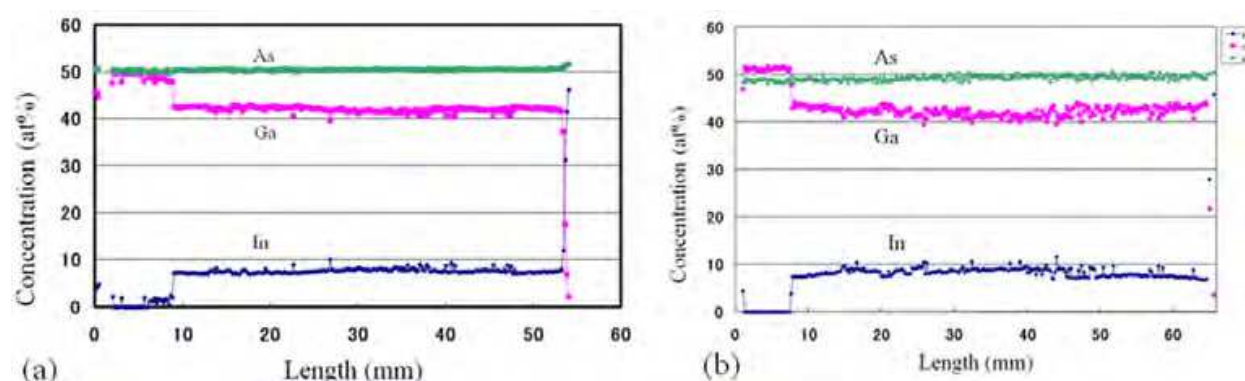


Fig. 14. Comparison of axial concentration profiles of crystals grown at different temperature gradient (a) $25^\circ\text{C}/\text{m}$ and (b) $37^\circ\text{C}/\text{cm}$. Average composition of both crystals is about $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$.

3.2.4 Temperature stability

Effect of temperature stability on compositional uniformity was investigated with average composition of $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ (Kinoshita et al., 2008). The most stable temperature was achieved when air flow in a furnace tube was shut. Temperature stability was $\pm 0.1^\circ\text{C}$ as shown in Fig. 15 (a). With air flow conditions, temperature stability got worse to $\pm 0.2^\circ\text{C}$ as shown in Fig. 15 (b). The stability difference was small but this small difference gave a considerable effect on the compositional uniformity as shown in Fig. 16. When the temperature stability was $\pm 0.1^\circ\text{C}$, InAs mole fraction was 0.13 with σ of 0.0005 where σ is the standard deviation in the distance between 20 and 40mm. When the temperature stability was $\pm 0.2^\circ\text{C}$, InAs concentration uniformity was 0.13 with σ of 0.006 in the distance between 20 and 40mm. The reason why the temperature fluctuation influence on the compositional uniformity may be related to the crystallization from the almost saturated melt, namely, temperature fluctuation gives rise to the formation of constitutionally supercooled region in a melt and such region crystallizes earlier, then it is not strange that earlier crystallized region has lower In concentration. Improvement in compositional stability resulted in higher crystal quality. X-ray rocking curve measurements showed that full width at half maximum (FWHM) of rocking curve ranges from 0.03 to 0.04 degrees for crystals grown at temperature stability $\pm 0.1^\circ\text{C}$. Such small FWHM shows good crystallineity which can be used as a substrate of laser diodes.

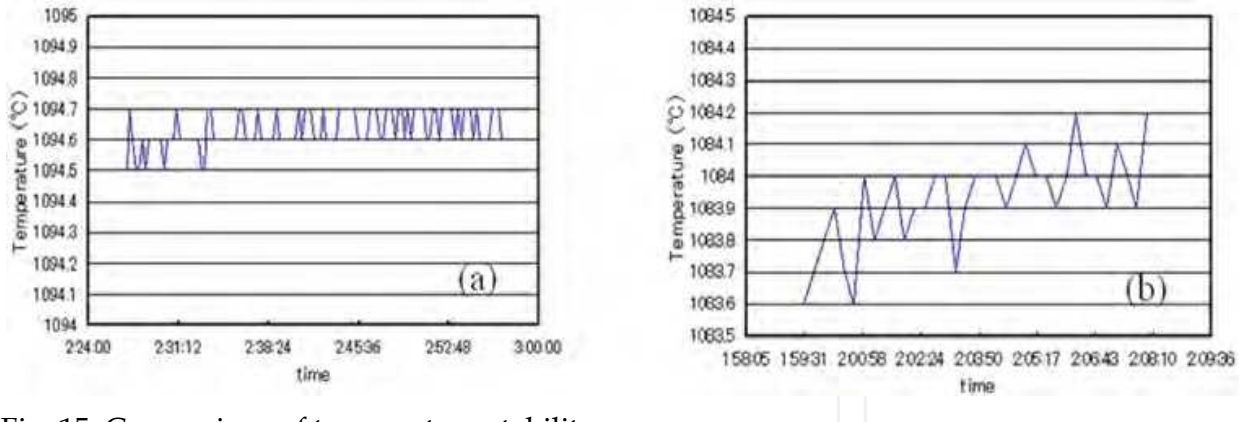


Fig. 15. Comparison of temperature stability.

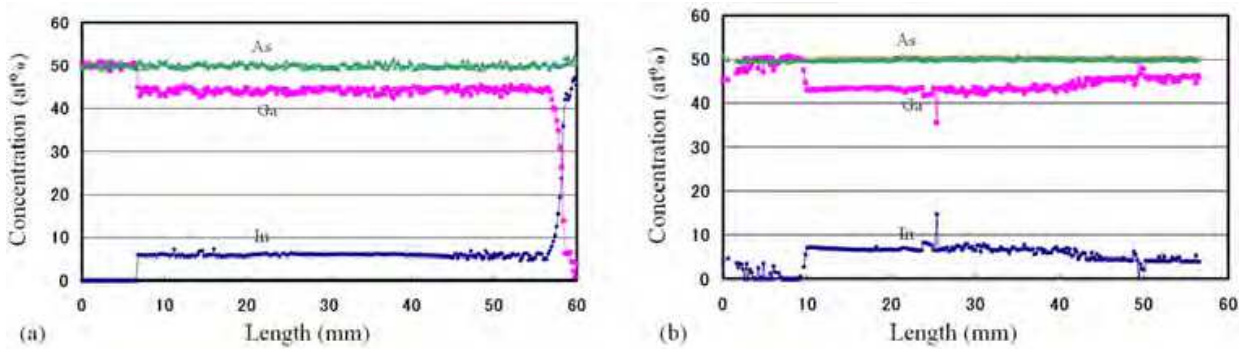


Fig. 16. Comparison of concentration profiles for two crystals grown at different temperature stability (a) $\pm 0.1^{\circ}\text{C}$ and (b) $\pm 0.2^{\circ}\text{C}$.

3.2.5 Characterization as substrates of laser diodes

Improvement in compositional stability resulted in higher crystal quality. Figure 17 shows X-ray rocking curves in the distance between 26 mm and 56 mm at an interval of 5mm for the crystal grown at the temperature stability of $\pm 0.1^{\circ}\text{C}$. The ω -scan around the (004) diffraction peak was detected by an open detector. Cu $k_{\alpha 1}$ radiation from an X-ray tube (loaded power; 40 kV, 30 mA) was monochromated by a four crystal Ge(220) monochromator and was incident on a crystal. The illuminated area was limited to about $1 \times 1 \text{ mm}^2$ by a divergence and a scatter slit. Full width at half maximum (FWHM) of rocking curve ranges from 0.03 to 0.04 degrees (from 108 to 144 arc seconds). Such small FWHM shows good crystallineity which can be used as a substrate of laser diodes. High quality region extended to $10 \times 30 \text{ mm}^2$. When InAs mole fraction increases, FWHM increased. This phenomenon is usual in many alloy crystals. When solute concentration increases, compositional uniformity is deteriorated and strain increases in the grown crystal. The increased strain degrades crystalline quality. Therefore, the lower InAs mole fraction, the higher is the quality of grown crystals. In the course of our study, we found that $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$ could be used as substrates of $\lambda = 1.3 \text{ }\mu\text{m}$ laser diodes instead of $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$. This is beneficial for ternary crystal growth since higher quality can be expected. Sn was doped as n-type dopant for substrate use. Carrier concentration was measured to be $5 - 8 \times 10^{18} \text{ cm}^{-3}$. Etch pit densities were in the range between 1×10^3 and $3 \times 10^4 \text{ cm}^{-2}$, which is sufficiently low for a substrate.

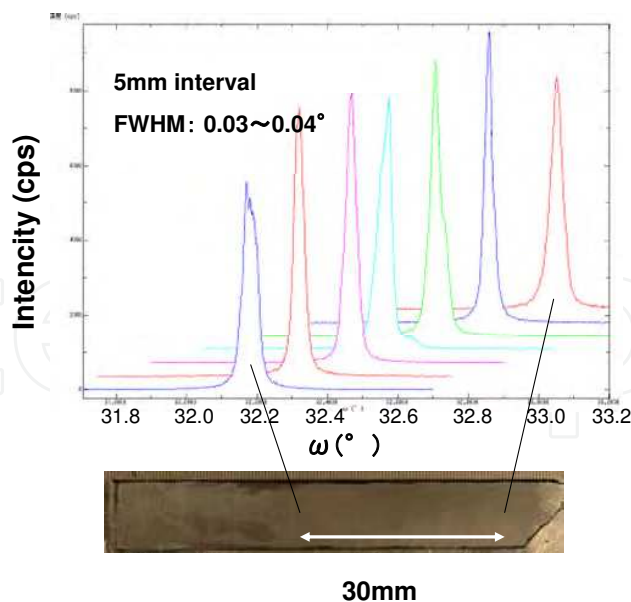


Fig. 17. FWHMs of X-ray rocking curves at seven positions. They range from 0.03 to 0.04° for the length of 30 mm.

3.3 Increase of a crystal width

Convection in a melt is suppressed in a thin melt. In platy crystal growth, width is considered to be independent of convection strength when thickness of crystals is limited. From this point of view, width of platy crystals was increased step by step from 10 mm to 20, 30, and 50 mm (Kinoshita et al., 2010). In all cases, thickness of platy crystals was set to be 2 mm. The most difficult point was to keep high temperatures in a feed area as growth ampoule was translated towards low temperature side. When width of platy crystals was increased, diameter of boron nitride (BN) crucible increased. BN has higher thermal conductivity than quartz and large diameter BN crucible transferred more heat to lower temperature side. For keeping high temperatures in a feed area, length of BN crucible was increased to receive more radiation from a heater.

Figure 18 shows an example of 30 mm wide platy crystal. Outlook of roughly polished surface is shown in Fig. 18 (a) and InAs mole fractions measured along a centre line and 10 mm away from the centre line are shown in Fig. 18 (b). Single crystalline area larger than $30 \times 30 \text{ mm}^2$ was obtained. InAs mole fractions were constant and 0.10 ± 0.01 was achieved for the distance of about 40 mm. Good crystallinity with the FWHM of less than 0.04° in X-ray rocking curves was obtained. Such excellent compositional uniformity and crystal quality owe to matching of sample translation rate and freezing rate and resulting in a fixed freezing interface position relative to heater segments. In this point of view, constant temperature gradient during translation of a sample was important for obtaining compositional uniformity. Such increase in surface area in platy crystals without deteriorating crystal quality also shows that convection in a melt is suppressed by the limitation of melt thickness (2 mm) and increase in melt width did not cause convection. If convection occurs in a melt, local inhomogeneity will be resulted due to stirring of a melt by convection. Good homogeneity shows that convection was suppressed in the course of crystal growth.

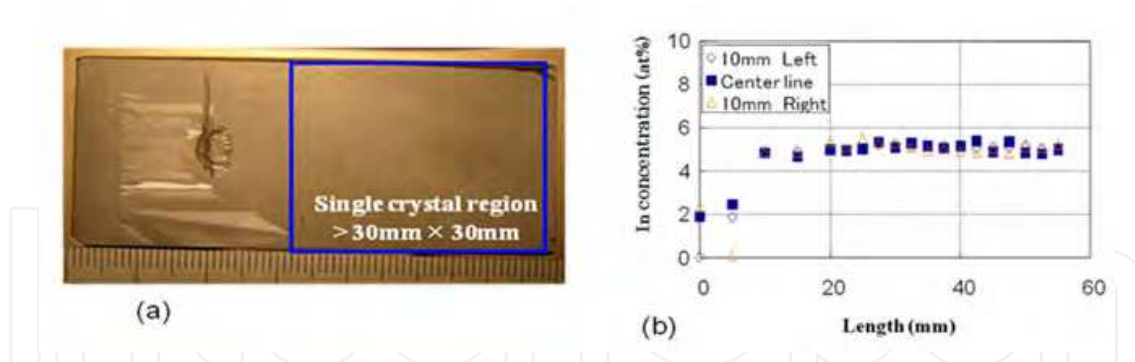


Fig. 18. An example of 30 mm wide platy crystal; (a) roughly polished surface of a crystal and (b) InAs mole fractions along growth axis.

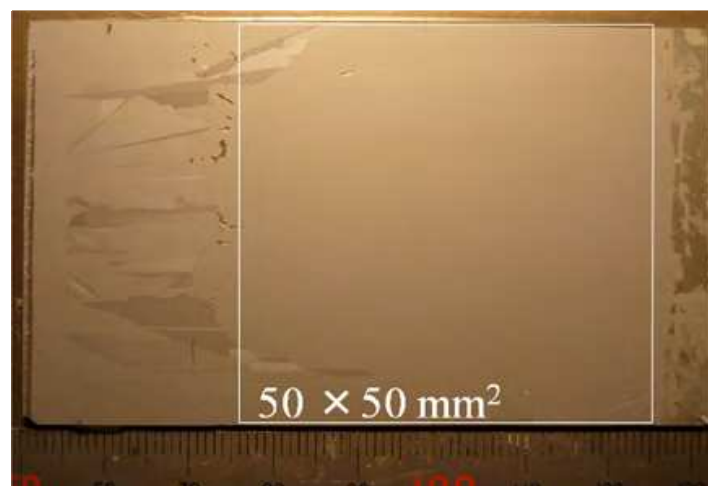


Fig. 19. An example of 50 mm wide crystal (Single crystalline area of almost $50 \times 50 \text{ mm}^2$ is obtained).

Now we have succeeded in growing 50 mm wide platy crystals. Figure 19 shows an example of 50 mm wide crystal. Single crystalline area of almost $50 \times 50 \text{ mm}^2$ is obtained. For mass production of laser diodes, large platy crystals with good reproducibility are required. As described earlier, crystals do not take over seed orientation but take over feed orientation. In this case, similar single crystallization mechanism works, too. Reproducibility is now more than 90%.

4. Laser diodes fabrication

Laser diodes with emitting wavelength $\lambda = 1.3 \text{ }\mu\text{m}$ were fabricated on $\text{In}_x\text{Ga}_{1-x}\text{As}$ ($x: 0.1 - 0.13$) substrates. Ishikawa (1993) demonstrated merits of a ternary substrate for high optical gain with small temperature dependence. In the course of study, we found that $\text{In}_x\text{Ga}_{1-x}\text{As}$ ($x: 0.1 - 0.13$) crystals can be used as substrates owing to development of thin film growth technology. Low InAs content crystals are beneficial to substrates because they have better thermal conductivity than high InAs content crystals.

4.1 Fabrication procedures

$\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ platy crystals were polished mechano-chemically to 0.5 mm thickness. Surface roughness of mirror polished substrates was measured to be less than several nano-meters. Strained multiple-quantum-wells (MQWs) having the combination of $\text{In}_{0.12}\text{Ga}_{0.88}\text{As}$ and $\text{In}_{0.38}\text{Ga}_{0.62}\text{As}$ as shown in Fig. 20 (a) were prepared by metal-organic vapor phase epitaxy (MOVPE) on the cleaned substrate (Arai et al., 2009). The active region was consisted of four $\text{In}_{0.38}\text{Ga}_{0.62}\text{As}$ wells and five $\text{In}_{0.12}\text{Ga}_{0.88}\text{As}$ barriers sandwiched by two separate-confinement heterostructure (SCH) layers. The stripe mesa type laser was fabricated by chemical etching. Cross sectional view of a fabricated mesa stripe is shown in Fig. 20 (b). The stripe mesa was in the [011] direction, forming a reverse mesa. Bottom ridge width of 1.7 μm enabled single mode lasing. Selective wet etching was utilized for such narrow ridge formation.

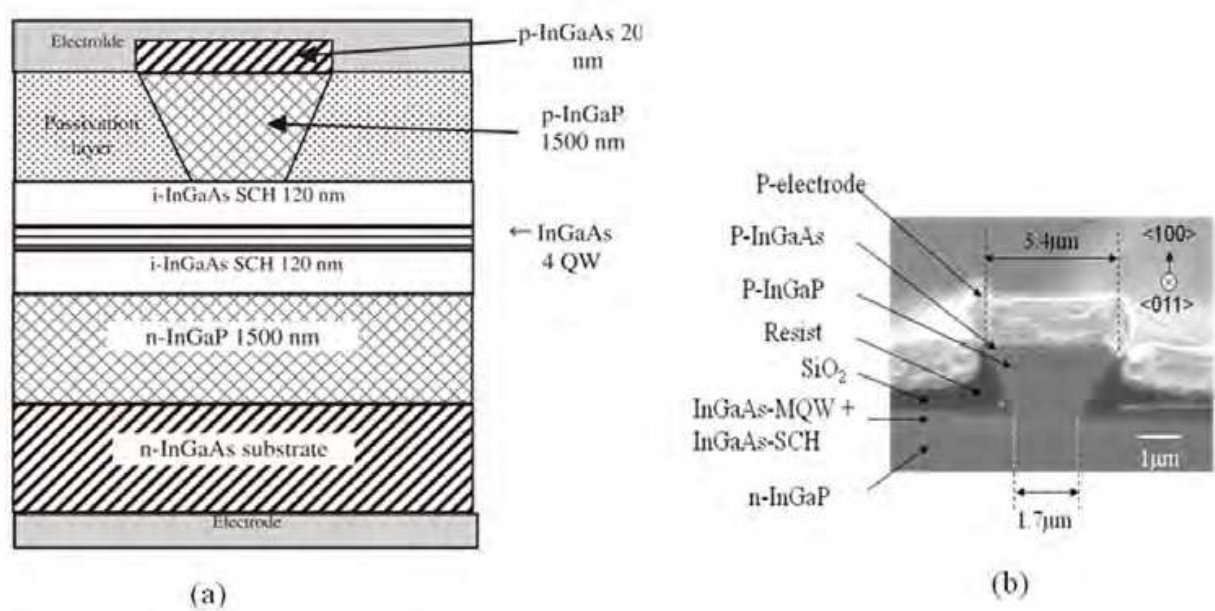


Fig. 20. Cross sectional view of a fabricated laser diode, (a) schematic drawing and (b) electron micrograph.

4.2 Characterization of laser diodes

Figure 21 (a) shows the bias current versus output power (I - L) characteristics of a fabricated laser diode. The threshold current is 7.2 mA. Figure 21 (b) is the lasing spectrum at a bias of 20 mA. The lasing wavelength is 1.31 μm . The laser exhibits a maximum operating temperature of 80 and 150 $^{\circ}\text{C}$ under CW and pulsed operation, respectively. Therefore, the limiting factor was self-heating. An effective way of overcoming this thermal problem is to reduce the indium content of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ substrate and to introduce InAlGaAs barrier layers. We therefore tried to realize 10 Gbps modulation over 85 $^{\circ}\text{C}$ by eliminating the thermal problems and we prepared an $\text{In}_{0.08}\text{Ga}_{0.92}\text{As}$ substrate. Laser diodes on an $\text{In}_{0.08}\text{Ga}_{0.92}\text{As}$ substrate showed improved lasing characteristics. Self heating problem due to bad thermal conductivity of $\text{In}_{0.13}\text{Ga}_{0.87}\text{As}$ substrates was settled by reducing InAs content in the substrate. However, emitting wavelength was 1.26 μm due allowance limit of InAs content in active layers. Figure 22 shows continuous wave (CW) lasing characteristics of this laser diode at various temperatures. CW operation was

possible up to 150°C and was much improved. Characteristic temperature of the threshold current density between 25 and 125°C was about 90 K and showed a higher value compared with that of 1.31 μm laser (about 70 K). Such high temperature stability in output power is also expected in 1.31 μm laser diodes by optimizing heat sink structure and heat sink materials.

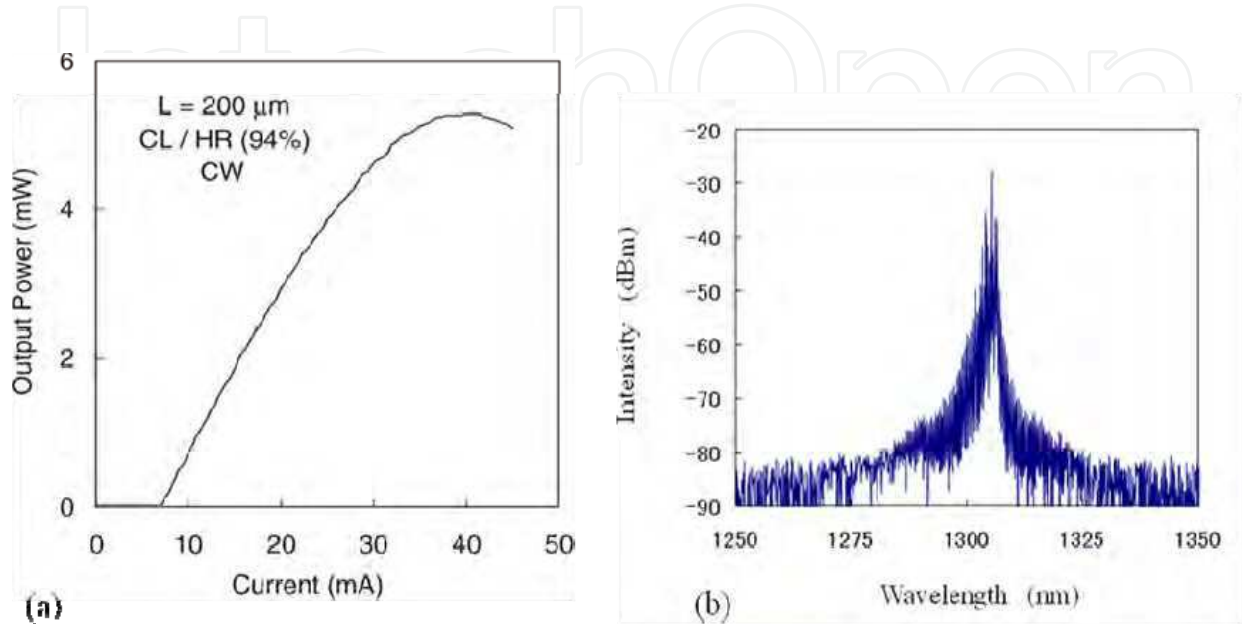


Fig. 21. Lasing characteristics of a fabricated laser diode. (b) is the lasing spectrum at a bias of 20 mA.

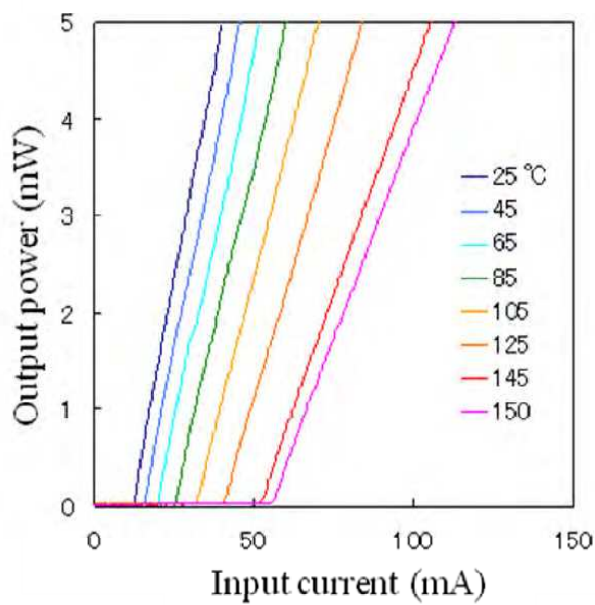


Fig. 22. CW lasing characteristics at various temperatures.

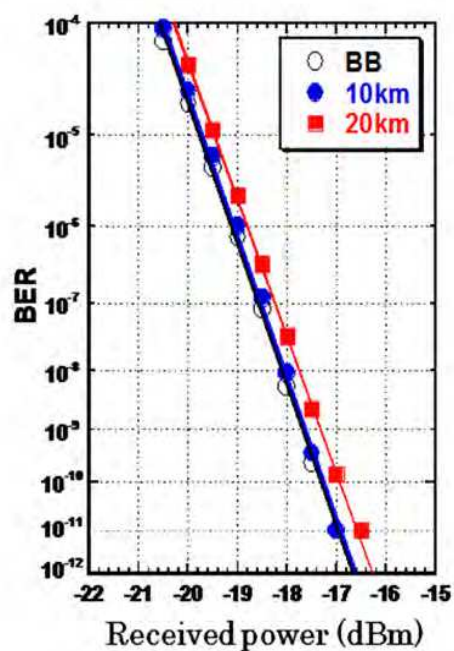


Fig. 23. Bit error rate after 20 km transmission.

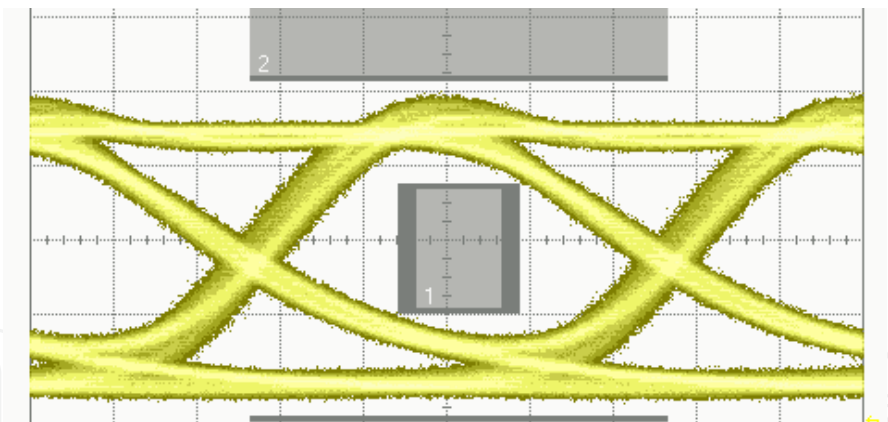


Fig. 24. Eye diagram at the modulation of 10 Gbps at 85°C.

A 10-Gbps direct modulation and transmission tests through a single mode fiber up to 20 km at 25°C were successfully performed using a fabricated laser diode with $\lambda = 1.31\text{ }\mu\text{m}$ as shown in Fig. 23. Bit error rate (BER) for back-to-back configuration after 20 km transmission was less than 10^{-7} at -18 dBm. We also tested 1.26 μm wavelength laser diodes. In this laser diodes, 10 Gbps modulation at 85°C was confirmed by an electrically filtered back-to-back (BB) eye diagram as shown in Fig. 24. The extinction ratio and the mask margin of the synchronous optical network (SONET) mask were 7 dB and 9%, respectively. These results show feasibility of fabricated laser diodes.

5. Conclusions

The TLZ method has been invented for growing compositionally uniform alloy crystals. The solute concentration gradient is controlled by applied temperature gradient utilizing saturation of solute in a zone. Owing to this merit, the spontaneous growth rate is calculated from the diffusion controlled steady-state growth conditions. Compositionally uniform alloy crystals are obtained merely by translating samples at the calculated growth rate relatively to a heater. Principle of the TLZ method was proved by the growth of 2 mm diameter crystals since convection in a melt is suppressed and diffusion limited growth is possible in capillary tubes even on the ground. As predicted, compositionally uniform crystals were grown without initial transient region which is typical to directional solidification method. Although the TLZ method is a superior method, it has limitations and they were also studied theoretically and experimentally. Limitation by temperature gradient, limitation by zone length, limitation by constitutional supercooling, and limitation by convection in a melt were made clear. In microgravity, TLZ growth is free from limitation by convection in a melt. In this point, microgravity is beneficial for TLZ growth. We grew platy $\text{In}_x\text{Ga}_{1-x}\text{As}$ (x : 0.08 – 0.13) crystals by the TLZ method on the ground for substrates of 1.3 μm wavelength laser diodes. In platy crystal growth, convection in a melt was suppressed by limiting thickness of platy crystals to 2 mm and large surface area was obtained by increasing width of platy crystals. Laser diodes on $\text{In}_x\text{Ga}_{1-x}\text{As}$ (x : 0.08 – 0.13) substrates showed excellent temperature stability in output power as predicted by Ishikawa et al. (1993). Now 50×50 mm² platy crystals were reproducibly grown and mass production of laser diodes with emitting wavelength of 1.3 μm is expected.

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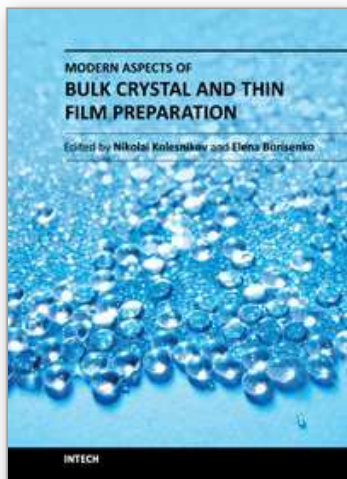
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